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TASK ANALYSIS OF STATE AND LOCAL AIR POLLUTION CONTROL AGENCIES AND DEVELOPMENT OF STAFFING GUIDELINES

VOLUME D. DETAILED TASK DATA, AND STAFF GUIDANCE. LABORATORY SUPPORT

APPLIED SCIENCE ASSOCIATES, INC.

PREPARED FOR
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Task Analysis of State and
Local Air Pollution Control Agencies and
Development of Staffing Guidelines

VOLUME D
Detailed Task Data, and Staff Guidance LABORATORY SUPPORT

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16. Abstracts Task data and staffing guidance are presented covering a group of tasks which are generally performed by a chemical analysis laboratory in support of the agency's air monitoring and source testing efforts. The tasks include standard chemical analyses, equipment maintenance, supervision, and development of new methods. The operations are performed by Chemists, Chemical Laboratory Technicians, and Equipment Technicians. The following tasks are included: (1) determination of nitrogen dioxide and nitric oxide concentrations in the atmosphere using the Saltzman Method; (2) determination of sulfur dioxide concentration in the atmosphere using the West-Gaeke Method; (3) determination of sulfur dioxide and sulfur trioxide concentrations in stack gases; (4) determination of suspended particulate concentration in the atmosphere by means of high volume sampling; (5) determination of hydrogen sulfide concentration in the atmosphere using the methylene blue method; (6) determination of nitrate concentration to suspended atmospheric particulates using the 2, 4 Xylenol Method; (7) determination of sulfate concentration in suspended atmospheric particulates using the turbidimetric barium sulfate method; (8) determination of metal concentration in suspended atmospheric particulates by means of high volume sampling; (9) determination of particulate concentration in stack emissions; (10) identification of the constituents of dust particles; (11) maintenance of laboratory devices; (12) supervision of laboratory support tasks; and (13) development of new methods for the analysis of air pollutants.

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Task Analysis of State and Local Air Pollution Control Agencies and Development of Staffing Guidelines

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VOLUME D

Detailed Task Data, and
Staffing Guidance
LABORATORY SUPPORT

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November 1972

This is not an official policy and standards document. The opinions, findings, and conclusions are those of the authors and not necessarily those of the United States Environmental Protection Agency. Every attempt has been made to represent the present state of the art as well as subject areas still under evaluation. Any mention of products, or organizations, does not constitute endorsement by the United States Environmental Protection Agency.

INTRODUCTION

One of the pressing problems in the air pollution control effort at Federal, state, and local levels is planning manpower requirements and developing manpower resources. Questions are being asked such as, how many people are needed, what kind of past experience and education should they have, how should their jobs be structured, what do they need to know to do their jobs, what special abilities do they need, and what kind of training should they receive to do their jobs? These questions are becoming increasingly meaningful as the control effort broadens with the creation of more and more local agencies and as existing agencies increase the scope and depth of their programs. Adequate answers are required if progress is to continue toward the goal of clean air.

In order to begin to answer questions relevant to manpower planning and development, a data base describing the tasks to be performed by control agency personnel and the skills and knowledge they must have to perform those tasks effectively must be available. Guidance concerning the use of the data base in making staffing decisions must be prepared. It is the purpose of this study to provide such a data base and the appropriate guidance.

A. Objectives

The objectives of this project were the following:

1. To identify as great a proportion as possible of the population of tasks currently being performed by air pollution control agency personnel at the state and local level throughout the country.
2. To describe the identified tasks in terms of component behaviors and the skills and knowledge required to perform those behaviors.
3. To identify and describe categories of air pollution control agency personnel who would perform the tasks mentioned above.

continued

4. To structure and communicate the data which resulted from achieving the above objectives in a form which could be used by agency management in planning and developing manpower resources.

B. General Project Overview

The project was performed in two phases. Phase I dealt with achieving the first two project objectives, and resulted in the development of a detailed data base describing the major tasks performed by agency personnel in terms of the procedural components of the tasks and the skills and knowledge required to perform them. Phase II dealt with achieving the last two major objectives, and resulted in production of a guidance document which integrates and structures data developed in Phase I and presents it in a form designed to assist agency manpower developers.

THIS IS VOLUME D
Additional books available are:

VOLUME A: Guidance and Supporting Information for Staffing and Training Decisions in an Air Pollution Control Agency - Introduction and Directions for Using These Guidelines

VOLUME B: Guidance and Supporting Information for Staffing and Training Decisions in an Air Pollution Control Agency - Engineering

VOLUME C: Guidance and Supporting Information for Staffing and Training Decisions in an Air Pollution Control Agency - Field Enforcement

VOLUME E: Guidance and Supporting Information for Staffing and Training Decisions in an Air Pollution Control Agency - Air Monitoring and Meteorological Support

VOLUME F: Guidance and Supporting Information for Staffing and Training Decisions in an Air Pollution Control Agency - Source Testing

VOLUME G: Guidance and Supporting Information for Staffing and Training Decisions in an Air Pollution Control Agency - Agency Management, Program Development, and Public Information Support

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LABORATORY SUPPORT

The task data and staffing guidance presented in this volume cover a group of tasks which are generally performed by a chemical analysis laboratory in support of the agency's air monitoring and source testing efforts. The tasks include standard chemical analyses, equipment maintenance, supervision, and development of new methods. The operations are performed by Chemists, Chemical Laboratory Technicians, and Equipment Technicians.

The following tasks are included and are located within the volume as indicated below:

1. Determination of Nitrogen Dioxide and Nitric Oxide Concentrations in the Atmosphere Using the Saltzman Method — Page D-4
2. Determination of Sulfur Dioxide Concentration in the Atmosphere Using the West-Gaeke Method — Page D-9
3. Determination of Sulfur Dioxide and Sulfur Trioxide Concentrations in Stack Gases — Page D-14
4. Determination of Suspended Particulate Concentration in the Atmosphere by Means of High Volume Sampling — Page D-18
5. Determination of Hydrogen Sulfide Concentration in the Atmosphere Using the Methylene Blue Method — Page D-21
6. Determination of Nitrate Concentration in Suspended Atmospheric Particulates Using the 2, 4 Xylenol Method — Page D-27

7. Determination of Sulfate Concentration in Suspended Atmospheric Particulates Using the Turbidimetric Barium Sulfate Method Page D-32

8. Determination of Metal Concentration in Suspended Atmospheric Particulates by Means of High Volume Sampling Page D-37

9. Determination of Particulate Concentration in Stack Emissions Page D-42

10. Identification of the Constituents of Dust Particles Page D-47

11. Maintenance of Laboratory Devices Page D-52

12. Supervision of Laboratory Support Tasks Page D-56

13. Development of New Methods for the Analysis of Air Pollutants Page D-65

Determination of Nitrogen Dioxide and Nitric Oxide Concentrations in the Atmosphere Using the Saltzman Method

Task Overview

The Saltzman Method for determining concentrations of nitrogen dioxide and nitric oxide is routinely performed in several agencies. It varies somewhat from the reference method contained in the Federal Register (Reference 1). However, the difference lies in the particular reagents used. The underlying principle is tne same.

The Saltzman Method is a colorimetric method. As a result, the procedure is very similar to that of the West-Gaeke Method for determining the concentration of sulfur dioxide and consists of the following outline of steps:

1. Preparation of the chemical reagents required for performance of the task (including putting the absorbing reagent in the fritted bubbler in preparation for sampling).
2. Analysis of the sample.
3. Calculation of the sulfur dioxide concentration.

The assembly and operation of the apparatus used for collecting the sample will be considered within Air Monitoring (see Volume E).

Occupational Category: Chemical Laboratory Technician

Task Description

The procedure for this method of determining nitrogen dioxide and nitric oxide concentrations is presented in usable form in Reference 3. An abbreviated procedure also appears in Reference 2. Where grab sample bottles are to be employed in sampling the preparation of the bottles is considered part of this task.

Skill Requirements

1. The ability to prepare reagents and handle caustic or otherwise dangerous chemicals without splattering acid, precipitating an explosion, or otherwise injuring personnel or damaging equipment.

2. Ability to accurately obtain definite volumes of solutions using apparatus such as a pipette, syringe, volumetric flask, or burette.

3. Ability to use an analytical balance to obtain an accurate weight of a dry reagent or filter. This includes the ability to:

   a. Zero the balance.

   b. Adjust sensitivity range without extreme changes which might damage the balance.

   c. Read and interpret the indicated mass.

4. Ability to assemble sampling apparatus with each component in proper sequence using butt-to-butt connections, tygon tubing, and silicone or florocarbon grease as appropriate to create a leak-proof assembly.

5. Ability to operate a colorimeter or spectrophotometer in order to obtain absorbance readings of sample and standard solutions. This includes the ability to correctly input sample to the instrument and recognize signs of instrument malfunction.

6. Ability to quickly and correctly solve an algebraic equation in several unknowns using a detailed step-by-step procedure. Such equations include:

   a. Calculating the concentration of a pollutant given the sample absorbance reading and the calibration curve.

   b. Converting the volume of gas sampled to the volume at standard temperature and pressure using the perfect gas equation.

7. Ability to prepare a calibration curve and compute the slope of the best fitting straight line, following a detailed step-by-step procedure. This may require the use of regression analysis by the least squares method.

8. Ability to read and interpret data from a table, psychometric chart, or a nomograph.

9. Ability to distill water so that it is deionized and nitrate-free.

10. Ability to measure the porosity of the fritted bubbler.

11. Ability to evacuate grab-sample bottles using a vacuum pump, manometer, and a three-way stopcock connection.

Knowledge Requirements

1. Knowledge of general chemical nomenclature in the following areas:

   a. The metric system of measurement

   b. Standard analytical laboratory apparatus (e.g., flasks, burettes, absorbers, pumps, etc.)

   c. Standard analytical laboratory instrumentation (e.g., colorimeter, spectrophotometer, rotameter, etc.)

   d. Standard chemical processes (e.g., filtration, titration, precipitation, etc.)

   e. Commonly used reagents

   f. Commonly measured pollutants

   g. Computations (e.g., standard temperature and pressure, calibration plot, line of best fit, etc.)

2. Knowledge of the composition of required reagents, the procedures for their preparation, the cautions attending preparation and use, method of their storage, and their shelf life.

3. Knowledge of the apparatus required for the task and how it is configured.

4. Knowledge of the task procedure with all steps in their proper sequence.

5. Knowledge of the procedure for operating a spectrophotometer or colorimeter.

6. Knowledge of the procedure for distilling deionized and nitrate-free water.

7. Knowledge of the procedure for operating and adjusting a laboratory balance.

8. Knowledge of the procedure for calculating the pollutant concentration including the correction to standard temperature and pressure as required.

9. Knowledge of the procedure for quantitatively diluting exposed absorbing reagent when the color is too dark to be read. This includes making appropriate adjustments to the pollutant concentration calculation.

References

1. Environmental Protection Agency. Reference method for the determination of nitrogen dioxide in the atmosphere (24 hour sampling method). Federal Register, Vol. 36, No. 84, Friday, April 30, 1971, pp. 8200-8201.

2. Jacobs, Morris B. The chemical analysis of air pollutants. New York: Interscience Publishers, Inc., 1970, pp. 209-215.

3. U. S. Department of Health, Education, and Welfare. Selected methods for the measurement of air pollutants. PHSP #999-AP-11, 1965, pp. C1-7.

Special Staffing Guidance

The following tasks also involve the use of the colorimeter (or spectrophotometer) and require similar skills and knowledge and so

may be efficiently performed by the same person who performs the task considered here:

1. The West Gaeke Analysis (see Page D-9).
2. The Hydrogen Sulfide Analysis (see Page D-21).
3. Sulfates Analysis (see Page D-32).
4. Nitrates Analysis (see Page D-27).

Determination of Sulfur Dioxide Concentration in the Atmosphere Using the West-Gaeke Method

Task Overview

There are various methods for measuring the concentration of sulfur dioxide in the atmosphere. These include:

1. West-Gaeke Method
2. Hydrogen Peroxide Method
3. Iodine-Thiosulfate Method
4. Iodine Method

These methods are described in References 1 through 4.

The West-Gaeke Method (sometimes called the Pararosaniline or Disulfitomercurate Method) has been put forth as a reference method in the Federal Register (Reference 1), and it is a method often used by air pollution control agencies. Briefly, the procedure consists of the following outline of steps:

1. Preparation of the chemical reagents required for performance of the task (including placing the absorbing reagent in the absorber in preparation for sampling).
2. Analysis of the sample.
3. Calculation of the sulfur dioxide concentration.

This task is concerned only with preparing for and conducting analysis of a sample. The assembly and operation of the apparatus used for collecting the sample will be considered within Air Monitoring (see Volume E).

Occupational Category: Chemical Laboratory Technician

Task Description

The procedure for this method of determining sulfur dioxide concentration is presented in usable form in Reference 3.

Skill Requirements

1. The ability to prepare reagents and handle caustic or otherwise dangerous chemicals without splattering acid, precipitating an explosion, or otherwise injuring personnel or damaging equipment.

2. Ability to accurately obtain definite volumes of solutions using apparatus such as a pipette, syringe, volumetric flask, or burette.

3. Ability to use an analytical balance to obtain an accurate weight of a dry reagent or filter. This includes the ability to

    a. Zero the balance.

    b. Adjust sensitivity range without extreme changes which might damage the balance.

    c. Read and interpret the indicated mass.

4. Ability to clean glassware and other apparatus without breakage or injury to oneself.

5. Ability to filter precipitate out of sample solution and wash the filter and apparatus so as to recover all of the filtrate.

6. Ability to operate a colorimeter or spectrophotometer in order to obtain absorbance readings of sample and standard solutions. This includes the ability to correctly input sample to the instrument and recognize signs of instrument malfunction.

7. Ability to quickly and correctly solve an algebraic equation in several unknowns using a detailed step-by-step procedure. Such equations include:

    a. Calculating the concentration of a pollutant given the sample absorbance reading and the calibration curve.

b. Converting the volume of gas samples to the volume at standard temperature and pressure using the perfect gas equation.

8. Ability to prepare a calibration curve and compute the slope of the best fitting straight line, following a detailed step-by-step procedure. This may require the use of regression analysis by the least squares method.

Knowledge Requirements

1. Knowledge of general chemical nomenclature in the following areas:
   a. The metric system of measurement
   b. Standard analytical laboratory apparatus (e.g., flasks, burettes, absorbers, pumps, etc.)
   c. Standard analytical laboratory instrumentation (e.g., colorimeter, spectrophotometer, rotameter, etc.)
   d. Standard chemical processes (e.g., filtration, titration, precipitation, etc.)
   e. Commonly used reagents
   f. Commonly measured pollutants
   g. Computations (e.g., standard temperature and pressure, calibration plot, line of best fit, etc.)
2. Knowledge of the composition of required reagents, the procedures for their preparation, the cautions attending preparation and use, method of their storage, and their shelf life.
3. Knowledge of the apparatus required for the task and how it is configured.
4. Knowledge of the highly poisonous nature of sodium tetrachloromercurate and the corrosive effects of acids.

5. Knowledge of the procedure for operating a spectrophotometer or colorimeter.

6. Knowledge of the procedure for estimating the purity of pararosaniline hydrochloride.

7. Knowledge of the procedure for operating and adjusting a laboratory balance.

8. Knowledge of the procedure for calculating the pollutant concentration including the correction to standard temperature and pressure as required.

References

1. Environmental Protection Agency. Reference method for the determination of sulfur dioxide in the atmosphere (pararosaniline method). Federal Register, Vol. 36, No. 84, Friday, April 30, 1971, pp. 8187-8190.

2. Jacobs, Morris B. The chemical analysis of air pollutants. New York: Interscience Publishers, Inc., 1970, pp. 170-178.

3. U. S. Department of Health, Education, and Welfare. Selected methods for the measurement of air pollutants. PHSP #999-AP-11, 1965, pp. A1-5.

4. West, P. W., and Gaeke, G. C. Fixation of sulfur dioxide and disulfitomercurate (II) and subsequent colorimetric estimation. Journal of Analytical Chemistry, Vol. 28, No. 12, December 1956, pp. 1816-1819.

Special Staffing Guidance

The following tasks also involve the use of the colorimeter (or spectrophotometer), require similar skills and knowledge and so may be efficiently performed by the same person who performs the task

considered here:

1. The Saltzman Analysis (see Page D-4).
2. The Hydrogen Sulfide Analysis (see Page D-21).
3. Sulfates Analysis (see Page D-32).
4. Nitrates Analysis (see Page D-27).

Determination of Sulfur Dioxide and Sulfur Trioxide Concentrations in Stack Gases

Task Overview

This is a method which was developed by the Shell Development Company (Reference 1), and it is suitable for measuring concentrations of sulfur dioxide and sulfur trioxide in stack gases. The following is an outline of the task procedure:

1. Preparation of the chemical reagents required for performance of the task (including putting the absorbing reagents in the absorbers).
2. Preparation of the sample for analysis.
3. Analysis of the sample using a titration technique.
4. Calculation of the concentrations.

The assembly and operations of the apparatus used to collect the stack gas sample will be covered under Source Testing (see Volume F).

Occupational Category: Chemical Laboratory Technician

Task Description

The procedure for determining sulfur dioxide and sulfur trioxide concentrations is presented in usable form in Reference 1.

Skill Requirements

1. The ability to prepare reagents and handle caustic or otherwise dangerous chemicals without splattering acid, precipitating an explosion, or otherwise injuring personnel or damaging equipment.
2. Ability to accurately obtain definite volumes of solutions using apparatus such as a pipette, syringe, volumetric flask, or burette.

3. Ability to use an analytical balance to obtain an accurate weight of a dry reagent or filter. This includes the ability to:

   a. Zero the balance.

   b. Adjust sensitivity range without extreme changes which might damage the balance.

   c. Read and interpret the indicated mass.

4. Ability to clean glassware and other apparatus without breakage or injury to oneself.

5. Ability to assemble sampling apparatus with each component in proper sequence using butt-to-butt connections, tygon tubing, and silicone or florocarbon grease as appropriate to create a leak-proof assembly.

6. Ability to filter precipitate out of sample solution and wash the filter and apparatus so as to recover all of the filtrate.

7. Ability to quickly and correctly solve an algebraic equation in several unknowns using a detailed step-by-step procedure. Such equations include:

   a. Calculating the concentration of a pollutant given the sample absorbance reading and the calibration curve.

   b. Converting the volume of gas sampled to the volume at standard temperature and pressure using the perfect gas equation.

8. Ability to distill water so that it is deionized and nitrate-free.

9. Ability to accurately titrate solutions and detect the subtle color change representing the end point.

10. Ability to create a partial vacuum in the isopropyl absorber and rinse the filter tube before suction is lost.

Knowledge Requirements

1. Knowledge of general chemical nomenclature in the following areas:
   a. The metric system of measurement
   b. Standard analytical laboratory apparatus (e.g., flasks, burettes, absorbers, pumps, etc.)
   c. Standard analytical laboratory instrumentation (e.g., colorimeter, spectrophotometer, rotameter, etc.)
   d. Standard chemical processes (e.g., filtration, titration, precipitation, etc.)
   e. Commonly used reagents
   f. Commonly measured pollutants
   g. Computations (e.g., standard temperature and pressure, calibration plot, line of best fit, etc.)
2. Knowledge of the composition of required reagents, the procedures for their preparation, the cautions attending preparation and use, method of their storage, and their shelf life.
3. Knowledge of the apparatus required for the task and how it is configured.
4. Knowledge of the task procedure with all steps in their proper sequence.
5. Knowledge of the procedure for distilling deionized and nitrate-free water.
6. Knowledge of the procedure for operating and adjusting a laboratory balance.
7. Knowledge of the procedure for calculating the pollutant concentration including the correction to standard temperature and pressure as required.

References

1. Shell Development Co., Anal. Dept. Shell development company method for determination of sulfur dioxide and sulfur trioxide. 4S16/59a. Emeryville, California: Author, 1959. pp. 85-87.

Special Staffing Guidance

Except for the need to perform a titration, the general skills and knowledge required to perform this task are similar to those required for the other standard wet test analyses. It can be assigned in combination with these other analyses as desired.

Determination of Suspended Particulate Concentration in the Atmosphere by Means of High Volume Sampling

Task Overview

The purpose of this task is to obtain an indication of the amount of solid particles suspended in the atmosphere. The task description below is representative of the way the task is performed in the agencies. It is also similar to the descriptions found in Reference 1 and 2. The actual collection of the sample using the high volume air sampler will be covered within Air Monitoring (see Volume E).

Occupational Category: Chemical Laboratory Technician or Equipment Technician

Task Description

Prepare filter:

1. Obtain high volume sample filter and dry in an oven for 24 hours. Cool filter in a desiccator.
2. Weigh the dry filter on an analytical balance.

Determine particulate concentration:

1. Dry the exposed filter for 24 hours; cool the filter; and obtain the new weight.
2. Calculate the total volume of air sampled by multiplying the average flow rate by the total sampling time.
3. Divide the particulate weight by the total volume of air sampled and express the concentration in ug/m^3.

Skill Requirements

1. Ability to use an analytical balance to obtain an accurate weight of a dry reagent or filter. This includes the ability to:
   a. Zero the balance.
   b. Adjust sensitivity range without extreme changes which might damage the balance.
   c. Read and interpret the indicated mass.

2. Ability to quickly and correctly solve an algebraic equation in several unknowns using a detailed step-by-step procedure. Such equations include:

    a. Calculating the concentration of a pollutant given the sample absorbance reading and the calibration curve.

    b. Converting the volume of gas measured to the volume at standard temperature and pressure using the perfect gas equation.

3. Ability to transport and handle filtering media using forceps if required so as not to contaminate it or lose material from it prior to weighing and analysis.

Knowledge Requirements

1. Knowledge of the apparatus required for the task and how it is configured.

2. Knowledge of the task procedure with all steps in their proper sequence.

3. Knowledge of the procedure for operating the drying oven and desiccator.

4. Knowledge of the procedure for operating and adjusting a laboratory balance.

5. Knowledge of the procedure for calculating the pollutant concentration including the correction to standard temperature and pressure as required.

References

1. Environmental Protection Agency. Reference method for the determination of suspended particulates in the atmosphere (high volume method). Federal Register, Vol. 36, No. 84, Friday, April 30, 1971, pp. 8191-2.

2. Jacobs, M. B. The chemical analysis of air pollutants, New York: Interscience Publishers, Inc., 1970, pp. 93-96.

Special Staffing Guidance

1. Because of the relative simplicity of this task it could effectively be performed by an inexperienced employee with a modest amount of on-the-job training.
2. Since this task does not require skills and knowledge necessary for wet test chemical analyses it could also be assigned to an Equipment Technician instead of the Chemical Laboratory Technician.
3. The following tasks also involve analysis of particulate matter as collected by the high volume air sampler and might also be assigned to the same person who performs this task:
   a. Metal Concentration Analysis (see D-37).
   b. Sulfates Analysis (see Page D-32).
   c. Nitrates Analysis (see Page D-27).

Determination of Hydrogen Sulfide Concentration in the Atmosphere Using the Methylene Blue Method

Task Overview

The Methylene Blue Method is the method typically used to measure the concentration of hydrogen sulfide in the atmosphere. Other methods available include the Cadmium Sulfide Method and the Impregnated Ceramic Tile Method.

The analysis procedure described below was derived from field observation and the description contained in Reference 1. The preparation and operation of the apparatus for collecting the sample is considered within Air Monitoring (see Volume E).

Occupational Category: Chemical Laboratory Technician

Task Description

Prepare the following reagents:

1. Absorbing Reagent. Dissolve 4.3 g of cadmium sulfate in water. Add 0.3 g of sodium hydroxide and 10 g of STRACTAN. Dilute the solution to one liter with freshly distilled water and mix.
2. Amine Acid Stock Solution. Add 50 ml of concentrated sulfuric acid to 30 ml of water and cool. Dissolve 12 g of N, N-dimethyl-p-phenylenediamine in the solution and refrigerate.
3. Amine Acid Working Solution. Dilute 25 ml of stock amine acid solution to one liter with 1:1 sulfuric acid and refrigerate.
4. Ferric Chloride Stock Solution. Dissolve 100 g of ferric chloride in water and dilute to 100 ml. Refrigerate.

5. Sodium Hydroxide Stock Solution, 1 Normal. Dissolve 40 g of sodium hydroxide in water and dilute to one liter.

6. Sodium Hydroxide Working Solution, 0.1 Normal. Dilute 100 ml of sodium hydroxide stock solution to one liter with water.

Prepare standard curve:

7. Purge a 100 ml, graduated Goetz tube with nitrogen gas and seal with a rubber septum. Using a syringe, add 100 ml of 0.1 N sodium hydroxide to the Goetz tube. Place the tube in cooling bath and cool to 4°C.

8. Withdraw 1.5 ml of hydrogen sulfide from a tank of purified hydrogen sulfide gas, using a syringe. Inject the hydrogen sulfide into the Goetz tube, vigorously shake, and cool again to 4°C. (This solution is stable for only 30 minutes. Therefore, it is required that two personnel complete the standardization process.).

9. Purge a 250 ml Erlenmeyer flask with nitrogen. Pipette 50 ml of the hydrogen sulfide solution to the flask. Then, add 10 ml of 0.01 N potassium iodate solution and about 100 mg of potassium iodide crystals. After the crystals have dissolved, add 2 ml of 2 N sulfuric acid. Mix the solution, stopper the flask, and set the flask in a cold water bath for 5 minutes.

10. Titrate the sulfide solution against a solution of 0.02 N sodium thiosulfate. Then, titrate distilled water against the same solution. Titrate the solutions to a pale yellow color; add a small amount of thyodine and titrate until clear.

11. Pipette 5 ml of standard hydrogen sulfide solution from the Goetz tube into a 50 ml volumetric flask. Dilute the solution to 50 ml with water. From this flask pipette 0, 1, 2, 3, 4, 5, 10, and 15 ml samples into 50 ml volumetric flasks with 30 ml of absorbing reagent each. To each flask add 0.6 ml of amine acid working solution and one drop of ferric chloride working solution, and dilute to 50 ml with water. Allow solution to stand for 30 minutes for color development.

12. Place each sample in a 10 cm tubular cell. Using a spectrophotometer (such as the Hitachi-Perkin-Elmer 130 U.V. Visible Spectrophotometer) obtain the absorbance reading for each sample at 670 mu.

13. Calculate the normality of the hydrogen sulfide from the results of the titration, using the equation: N x ml (thiosulfate) = N x ml (hydrogen sulfide). Convert the result to ug/ml and correct for the 1 to 10 dilution.

14. Plot a standard curve of absorbance vs. ug/ml of hydrogen sulfide. Compute the slope of the best fitting straight line.

Analyze air sample:

15. Add 0.6 ml of amine acid working solution and one drop of ferric chloride working solution to the sample. Dilute the sample to 50 ml with water in a volumetric flask. Also, prepare a blank using 30 ml of absorbing reagent, 0.6 ml of amine acid working solution, and one drop of ferric chloride working solution. (This is used for zeroing the spectrophotometer.) Allow the solutions to stand for 30 minutes.

16. Obtain the absorbance of the sample, using the spectrophotometer.

17. Determine the amount (ug) of hydrogen sulfide in the sample, using the absorbance reading and the standard curve. Calculate the volume of the sample from the sampling rate and time. Calculate the concentration of hydrogen sulfide in parts-per-billion.

Skill Requirements

1. The ability to prepare reagents and handle caustic or otherwise dangerous chemicals without splattering acid, precipitating an explosion, or otherwise injuring personnel or damaging equipment.

2. Ability to accurately obtain definite volumes of solutions using apparatus such as a pipette, syringe, volumetric flask, or burette.

3. Ability to use an analytical balance to obtain an accurate weight of a dry reagent or filter. This includes the ability to:

    a. Zero the balance.

    b. Adjust sensitivity range without extreme changes which might damage the balance.

    c. Read and interpret the indicated mass.

4. Ability to clean glassware and other apparatus without breakage or injury to oneself.

5. Ability to operate a colorimeter or spectrophotometer in order to obtain absorbance readings of sample and standard solutions. This includes the ability to correctly input sample to the instrument and recognize signs of instrument malfunction.

6. Ability to quickly and correctly solve an algebraic equation in several unknowns using a detailed step-by-step procedure. Such equations include:

    a. Calculating the concentration of a pollutant given the sample absorbance reading and the calibration curve.

    b. Converting the volume of gas sampled to the volume at standard temperature and pressure using the perfect gas equation.

7. Ability to prepare a calibration curve and compute the slope of the best fitting straight line, following a detailed step-by-step procedure. This may require the use of regression analysis by the least squares method.

8. Ability to read and interpret data from a table, psychometric chart, or a nomograph.

9. Ability to distill water so that it is deionized and nitrate-free.

10. Ability to handle pressurized gas without endangering personnel or equipment.

11. Ability to proficiently purge and seal a Goetz tube or Erlenmeyer flask with nitrogen gas.

12. Ability to accurately titrate solutions and detect the subtle color change representing the end point.

Knowledge Requirements

1. Knowledge of general chemical nomenclature in the following areas:

    a. The metric system of measurement.

    b. Standard analytical laboratory apparatus (e.g., flasks, burettes, absorbers, pumps, etc.).

    c. Standard analytical laboratory instrumentation (e.g., colorimeter, spectrophotometer, rotameter, etc.).

    d. Standard chemical processes (e.g., filtration, titration, precipitation, etc.).

    e. Commonly used reagents.

    f. Commonly measured pollutants.

    g. Computations (e.g., standard temperature and pressure, calibration plot, line of best fit, etc.).

2. Knowledge of the composition of required reagents, the procedures for their preparation, the cautions attending preparation and use, method of their storage, and their shelf life.

3. Knowledge of the apparatus required for the task and how it is configured.

4. Knowledge of task procedure with all steps in their proper sequence.

5. Knowledge of the procedure for operating a spectrophotometer or colorimeter.

6. Knowledge of the procedure for distilling deionized and nitrate-free water.

7. Knowledge of the procedure for operating and adjusting a laboratory balance.

8. Knowledge of the procedure for calculating the pollutant concentration including the correction to standard temperature and pressure as required.

References

1. Jacobs, M. B. The chemical analysis of air pollutants. New York: Interscience Publishers, Inc. 1970, pp. 182-188.

Special Staffing Guidance

The following tasks also involve the use of the colorimeter (or spectrophotometer) and require similar skills and knowledge and so may be efficiently performed by the same person who performs the task considered here:

a. The West-Gaeke Analysis (see Page D-9).

b. The Saltzman Analysis (see Page D-4).

c. Sulfates Analysis (see Page D-32).

d. Nitrates Analysis (see Page D-27).

Determination of Nitrate Concentration in Suspended Atmospheric Particulates Using the 2, 4 Xylenol Method

Task Overview

This task is very similar to the determination of sulfates in suspended atmospheric particulates and it is also performed routinely in many agencies. The task involves obtaining a 2, 4 xylenol treated water sample from a high volume air sampler filter. The Nitrated 2, 4 xylenol is extracted from the sample, purified, and analyzed colorimetrically. The analysis can be outlined as follows:

1. Preparation of the chemical reagents required for performance of the task.
2. Assembly of the refluxing apparatus.
3. Preparation of the sample for analysis.
4. Colorimetric analysis of the sample.
5. Preparation of standardized curve.
6. Calculation of the nitrate concentration.

The actual collection of the sample using the high volume air sampler will be covered within Air Monitoring (see Volume E).

Occupational Category: Chemical Laboratory Technician

Task Description

The procedure for this method of determining nitrate concentration is presented in usable form in Reference 2. An abbreviated description appears in Reference 1.

Skill Requirements

1. The ability to prepare reagents and handle caustic or otherwise dangerous chemicals without splattering acid, precipitating an explosion, or otherwise injuring personnel or damaging equipment.

2. Ability to accurately obtain definite volumes of solutions using apparatus such as a pipette, syringe, volumetric flask, or burette.

3. Ability to use an analytical balance to obtain an accurate weight of a dry reagent or filter. This includes the ability to:

    a. Zero the balance.

    b. Adjust sensitivity range without extreme changes which might damage the balance.

    c. Read and interpret the indicated mass.

4. Ability to clean glassware and other apparatus without breakage or injury to oneself.

5. Ability to assemble sampling apparatus with each component in proper sequence using butt-to-butt connections, tygon tubing, and silicone or florocarbon grease as appropriate to create a leak-proof assembly.

6. Ability to filter precipitate out of sample solution and wash the filter and apparatus so as to recover all of the filtrate.

7. Ability to operate a colorimeter or spectrophotometer in order to obtain absorbance readings of sample and standard solutions. This includes the ability to correctly input sample to the instrument and recognize signs of instrument malfunction.

8. Ability to quickly and correctly solve an algebraic equation in several unknowns using a detailed step-by-step procedure. Such equations include:

    a. Calculating the concentration of a pollutant given the sample absorbance reading and the calibration curve.

    b. Converting the volume of gas sampled to the volume at standard temperature and pressure using the perfect gas equation.

9. Ability to prepare a calibration curve and compute the slope of the best fitting straight line, following a detailed step-by-step procedure. This may require the use of regression analysis by the least squares method.

10. Ability to read and interpret data from a table, psychometric chart, or a nomograph.

11. Ability to transport and handle filtering media using forceps if required so as not to contaminate it or lose material from it prior to weighing and analysis.

12. Ability to draw off the various layers of a separated solution.

13. Ability to prevent the formation of emulsions in the sample by means of proper agitation.

Knowledge Requirements

1. Knowledge of general chemical nomenclature in the following areas:

    a. The metric system of measurement.

    b. Standard analytical laboratory apparatus (e.g., flasks, burettes, absorbers, pumps, etc.)

    c. Standard analytical laboratory instrumentation (e.g., colorimeter, spectrophotometer, rotameter, etc.)

    d. Standard chemical processes (e.g., filtration, titration, precipitation, etc.)

    e. Commonly used reagents

    f. Commonly measured pollutants

    g. Computations (e.g., standard temperature and pressure, calibration plot, line of best fit, etc.)

2. Knowledge of the composition of required reagents, the procedures for their preparation, the cautions attending preparation and use, method of their storage, and their shelf life.

3. Knowledge of the apparatus required for the task and how it is configured.

4. Knowledge of the task procedure with all steps in their proper sequence.

5. Knowledge of the procedure for operating a spectrophotometer or colorimeter.

6. Knowledge of the procedure for operating the drying oven and desiccator.

7. Knowledge of the procedure for operating the refluxing apparatus.

8. Knowledge of the procedure for operating and adjusting a laboratory balance.

9. Knowledge of the procedure for calculating the pollutant concentration including the correction to standard temperature and pressure as required.

10. Knowledge of the procedure for removing or adjusting for nitrates pre-existing on the filter medium.

References

1. Jacobs, M. B. The chemical analysis of air pollutants. New York: Interscience Publishers, Inc., 1970, pp. 117-118.

2. U. S. Department of Health, Education, and Welfare. Selected methods for the measurement of air pollutants. PHSP #999-AP-11, 1965, pp. J1-4.

Special Staffing Guidance

1. The following tasks also involve the use of the colorimeter (or spectrophotometer) and require similar skills and knowledge and so may be efficiently performed by the same person who performs the task considered here:

   a. The West-Gaeke Analysis (see Page D-9).

   b. The Saltzman Analysis (see Page D-4).

   c. The Hydrogen Sulfide Analysis (see Page D-21)

   d. Sulfates Analysis (see Page D-32).

2. The following tasks also involve analysis of particulate matter as collected by the high volume air sampler and might also be assigned to the same person who performs this task:

   a. Suspended Particulate Concentration Analysis (see Page D-42).

   b. Metal Concentration Analysis (see Page D-37).

   c. Sulfates Analysis (see Page D-32).

3. Because of the similarity between this task and the Sulfates Analysis (see D-32), both could be performed by the same person.

Determination of Sulfate Concentration in Suspended Atmospheric Particulates Using the Turbidimetric Barium Sulfate Method

Task Overview

This task is routinely performed in several agencies and involves colorimetric analysis of a barium chloride treated water sample obtained from a high volume air sampler filter. The outline of the procedure is as follows:

1. Preparation of the chemical reagents required for performance of the task.
2. Assembly of the refluxing apparatus.
3. Preparation of the sample for analysis.
4. Colorimetric analysis of the sample.
5. Preparation of standardized curve.
6. Calculation of the sulfate concentration.

The actual collection of the sample using the high volume air sampler will be covered within Air Monitoring (see Volume E).

Occupational Category: Chemical Laboratory Technician

Task Description

The procedure for this method of determining sulfate concentration is presented in usable form in Reference 2. An abbreviated description appears in Reference 1.

Skill Requirements

1. The ability to prepare reagents and handle caustic or otherwise dangerous chemicals without splattering acid, precipitating an explosion, or otherwise injuring personnel or damaging equipment.

2. Ability to accurately obtain definite volumes of solutions using apparatus such as a pipette, syringe, volumetric flask, or burette.

3. Ability to use an analytical balance to obtain an accurate weight of a dry reagent or filter. This includes the ability to:

    a. Zero the balance.

    b. Adjust sensitivity range without extreme changes which might damage the balance.

    c. Read and interpret the indicated mass.

4. Ability to clean glassware and other apparatus without breakage or injury to oneself.

5. Ability to assemble sampling apparatus with each component in proper sequence using butt-to-butt connections, tygon tubing, and silicone or florocarbon grease as appropriate to create a leak-proof assembly.

6. Ability to filter precipitate out of sample solution and wash the filter and apparatus so as to recover all of the filtrate.

7. Ability to operate a colorimeter or spectrophotometer in order to obtain absorbance readings of sample and standard solutions. This includes the ability to correctly input sample to the instrument and recognize signs of instrument malfunction.

8. Ability to quickly and correctly solve an algebraic equation in several unknowns using a detailed step-by-step procedure. Such equations include:

    a. Calculating the concentration of a pollutant given the sample absorbance reading and the calibration curve.

    b. Converting the volume of gas sampled to the volume at standard temperature and pressure using the perfect gas equation.

9. Ability to prepare a calibration curve and compute the slope of the best fitting straight line, following a detailed step-by-step procedure. This may require the use of regression analysis by the least squares method.

10. Ability to read and interpret data from a table, psychometric chart, or a nomograph.

11. Ability to transport and handle filtering media using forceps if required so as not to contaminate it or lose material from it prior to weighing and analysis.

Knowledge Requirements

1. Knowledge of general chemical nomenclature in the following areas:

    a. The metric system of measurement

    b. Standard analytical laboratory apparatus (e.g., flasks, burettes, absorbers, pumps, etc.)

    c. Standard analytical laboratory instrumentation (e.g., colorimeter, spectrophotometer, rotameter, etc.)

    d. Standard chemical processes (e.g., filtration, titration, precipitation, etc.)

    e. Commonly used reagents

    f. Commonly measured pollutants

    g. Computations (e.g., standard temperature and pressure, calibration plot, line of best fit, etc.)

2. Knowledge of the composition of required reagents, the procedures for their preparation, the cautions attending preparation and use, method of their storage, and their shelf life.

3. Knowledge of the apparatus required for the task and how it is configured.

4. Knowledge of the task procedure with all steps in their proper sequence.

5. Knowledge of the procedure for operating a spectrophotometer or colorimeter.

6. Knowledge of the procedure for operating the refluxing apparatus.

7. Knowledge of the procedure for operating and adjusting a laboratory balance.

8. Knowledge of the procedure for calculating the pollutant concentration including the correction to standard temperature and pressure as required.

9. Knowledge of the procedure for removing or adjusting for sulfates pre-existing on the filter medium.

References

1. Jacobs, M. B. The chemical analysis of air pollutants. New York: Interscience Publishers, Inc., 1970, pp. 116-117.

2. U. S. Department of Health, Education, and Welfare. Selected methods for the measurement of air pollutants. PHSP #999-AP-11, 1965, pp. I1-4.

Special Staffing Guidance

1. The following tasks also involve the use of the colorimeter (or spectrophotometer) and require similar skills and knowledge and so may be efficiently performed by the same person who performs the task considered here:

   a. The West-Gaeke Analysis (see Page D-9).

   b. The Saltzman Analysis (see Page D-4).

   c. The Hydrogen Sulfide Analysis (see Page D-21).

   d. The Nitrates Analysis (see Page D-27).

2. The following tasks also involve analysis of particulate matter as collected by the high volume air sampler and might be assigned to the same person who performs this task:

   a. Suspended Particulate Concentration Analysis (see Page D-18).

   b. Metal Concentration Analysis (see Page D-37).

   c. Nitrates Analysis (see Page D-27).

3. Because of the similarity between this task and the Nitrates Analysis (see D-27), both could be performed by the same person.

Determination of Metal Concentration in Suspended Atmospheric Particulates by Means of High Volume Sampling

Task Overview

The task described below is representative of various methods for measuring the concentration of metals in the suspended atmospheric particulates from a high volume sample. The method is based on the use of an atomic absorption spectrophotometer. Other methods include the use of an emission spectrograph or a photofluorimeter. Each method is based on specific atomic properties whose characteristics vary among different types of atoms. The task as described below is concerned with the analysis of a sample. The actual collection of the sample using the high volume air sampler will be covered within Air Monitoring (see Volume E).

Occupational Category: Chemical Laboratory Technician

Task Description

1. Take an aliquot of the sample filter and place it in an extraction cup.
2. Prepare a 20 ml solution of acid in an extracting flask (four parts, by volume, of 1:1 nitric acid to one part of 1:1 hydrochloric acid).
3. Assemble refluxing apparatus.
   a. Condenser.
   b. Extraction cup.
   c. Extracting Flask.
   d. Hot plate.

   Reflux the acid solution for two hours.
4. Add an additional 15 ml of acid and reflux for another two hours.

5. Allow the assembly to cool overnight.

6. Prepare vacuum-distillation apparatus:

    a. Pump.

    b. Primary and secondary traps.

    c. Extracting flask.

    d. Warm water bath.

7. Run distillation to completion, periodically replenishing dry ice in traps and warm water in the bath.

8. Remove the extraction flask from the apparatus.

9. Prepare one liter of acid solution (43 ml of concentrated hydrochloric aoid to 33 ml of concentrated nitric acid, diluted to one liter with water) and add 40 ml of the solution to the sample in the extraction flask.

10. Swirl the suspension in the flask. Cover the flask with parafilm and allow it to sit overnight.

11. Transfer the sample to a volumetric flask and dilute it to 100 ml with water.

12. Prepare four standard solutions by adding graduated amounts of metal salts (known concentration) to 8 ml aliquots of the sample.

13. Prepare atomic absorption spectrophotometer for the specific metal.

14. Take three absorbance readings on each standard solution and calculate the average.

15. Plot the absorbance against the concentration of metal added in each standard.

16. Construct a best fitting straight line and calculate the slope of the line using regression analysis by the least squares method.

17. Calculate the X-intercept of the curve and correct it, using the value obtained on a blank filter.

18. Obtain the volume of air sampled and compute the ambient concentration of the metal in ug/m^3.

19. Obtain the average residue concentration of the particulate and calculate the weight percentage of metal in the average residue.

Skill Requirements

1. The ability to prepare reagents and handle caustic or otherwise dangerous chemicals without splattering acid, precipitating an explosion, or otherwise injuring personnel or damaging equipment.

2. Ability to accurately obtain definite volumes of solutions using apparatus such as a pipette, syringe, volumetric flask, or burette.

3. Ability to clean glassware and other apparatus without breakage or injury to oneself.

4. Ability to assemble sampling apparatus with each component in proper sequence using butt-to-butt connections, tygon tubing, and silicone or florocarbon grease as appropriate to create a leak-proof assembly.

5. Ability to quickly and correctly solve an algebraic equation in several unknowns using a detailed step-by-step procedure. Such equations include:

    a. Calculating the concentration of a pollutant given the sample absorbance reading and the calibration curve.

    b. Converting the volume of gas sampled to the volume at standard temperature and pressure using the perfect gas equation.

6. Ability to prepare a calibration curve and compute the slope of the best fitting straight line, following a detailed step-by-step procedure. This may require the use of regression analysis by the least squares method.

7. Ability to transport and handle filtering media using forceps if required so as not to contaminate it or lose material from it prior to weighing and analysis.

8. Ability to insert a Gooch crucible into a vacuum-jacketed extractor without damaging the extractor.

9. Ability to operate the atomic absorption spectrophotometer so as to obtain accurate absorption readings of samples and standards. This includes the ability to correctly input sample to the instrument and to detect instrument malfunction.

10. Ability to perform a quantitative transfer of a solution from one container to another without losing any of the sample.

11. Ability to clean grease from the neck of a flask without contaminating the contained sample with the cleaning agent.

Knowledge Requirements

1. Knowledge of general chemical nomenclature in the following areas:

   a. The metric system of measurement.

   b. Standard analytical laboratory apparatus (e.g., flasks, burettes, absorbers, pumps, etc.)

   c. Standard analytical laboratory instrumentation (e.g., colorimeter, spectrophotometer, rotameter, etc.)

   d. Standard chemical processes (e.g., filtration, titration, precipitation, etc.).

   e. Commonly used reagents.

   f. Commonly measured pollutants.

   g. Computations (e.g., standard temperature and pressure, calibration plot, line of best fit, etc.).

2. Knowledge of the composition of required reagents, the procedures for their preparation, the cautions attending preparation and use, method of their storage, and their shelf life.

3. Knowledge of the apparatus required for the task and how it is configured.

4. Knowledge of the task procedure with all steps in their proper sequence.

5. Knowledge of the procedure for operating the refluxing apparatus.

6. Knowledge of the procedure for operating the vacuum distillation. apparatus.

7. Knowledge of the procedure for operating the atomic absorption spectrophotometer.

References

1. Jacobs, M. B. The chemical analysis of air pollutants. New York: Interscience Publishers, Inc., 1970, pp. 98-101.

Special Staffing Guidance

The following tasks also involve analysis of particulate matter as collected by the high volume air sampler and might also be assigned to the same person who performs this task:

1. Suspended Particulate Concentration Analysis (see Page D-18).

2. Sulfates Analysis (see Page D-32).

3. Nitrates Analysis (see Page D-27).

Determination of Particulate Concentration in Stack Emissions

Task Overview

The lab support requirement for determining the particulate concentration in stack emission depends on what method of particulate collection is employed. In general the analysis procedure involves the following steps:

1. Drying, weighing, and otherwise preparing the alundum or paper thimble filter, cyclone collector or electrostatic precipitator to be used in the sampling train.
2. Adding measured amounts of water to the impingers to be used in the sampling train.
3. Determining the particulate concentration of the dry collector and impingers after sampling.

The assembly and operation of the sampling train used for collecting the particulate sample will be covered under Source Testing (see Volume F).

Occupational Category: Chemical Laboratory Technician

Task Description

Procedures covering the various techniques for analysis of particulate concentration is provided in references 1, 2, 4, 5, 6, 7, 10, and 11. The remainder of the publications listed (References 3, 8, and 9) contain useful information with regard to stack testing equipment and selected testing techniques.

Skill Requirements

1. Ability to accurately obtain definite volumes of solutions using apparatus such as a pipette, syringe, volumetric flask, or burette.

2. Ability to use an analytical balance to obtain an accurate weight of a dry reagent or filter. This includes the ability to:

    a. Zero the balance.

    b. Adjust sensitivity range without extreme changes which might damage the balance.

    c. Read and interpret the indicated mass.

3. Ability to clean glassware and other apparatus without breakage or injury to oneself.

4. Ability to assemble sampling apparatus with each component in proper sequence using butt-to-butt connections, tygon tubing, and silicone of florocarbon grease as appropriate to create a leak-proof assembly.

5. Ability to filter precipitate out of sample solution and wash the filter and apparatus so as to recover all of the filtrate.

6. Ability to quickly and correctly solve an algebraic equation in several unknowns using a detailed step-by-step procedure. Such equations include:

    a. Calculating the concentration of a pollutant given the sample absorbance reading and the calibration curve.

    b. Converting the volume of gas sampled to the volume at standard temperature and pressure using the perfect gas equation.

7. Ability to sand the lip of a alundum thimble to assure a good seal, removing all loose material.

Knowledge Requirements

1. Knowledge of the apparatus required for the task and how it is configured.

2. Knowledge of the task procedure with all steps in their proper sequence.

3. Knowledge of the procedure for operating the drying oven and desiccator.

4. Knowledge of the procedure for operating and adjusting a laboratory balance.

5. Knowledge of the procedure for obtaining the particulate weight trapped by both the dry collector and the impingers and combining these to obtain the total particulate weight.

6. Knowledge of the procedure for calculating:

    a. Gas volume, corrected to standard conditions of temperature and pressure.

    b. Particulate concentration.

    c. Emission rate.

7. Knowledge of procedure for draining the sampling train of moisture and measuring the condensate.

References

1. American Society for Testing and Materials. Standard method for sampling stacks for particulate matter. Philadelphia: Author, 1971. D2928-71.

2. American Society of Mechanical Engineers. Determining dust concentration in a gas stream. New York: Author, 1957.

3. Committee on Industrial Ventilation. Industrial ventilation. A manual of recommended practice. Lansing, Michigan: American Conference of Governmental Industrial Hygienists, 1970 (11th Edition). pp. 9(1-8).

4. Devorkin, H., Chass, R., Fudurich, A., & Kanter, C. R. Holmes (Ed.) Air pollution source testing manual. Los Angeles: Air Pollution Control District, Los Angeles County, November 1965.

5. Environmental Protection Agency. Standards of performance for new stationary sources. Federal Register, Volume 36, Number 159, August 17, 1971.

6. Haaland, H. H. (Ed.) Methods for determination of velocity, dust and mist content of gases. Bulletin WP-50. Seventh Edition. Los Angeles: Western Precipitation Division/Joy Manufacturing Company, 1968.

7. Jacobs, M. B. The chemical analysis of air pollutants. New York: Interscience Publishers, Inc., 1960. pp. 140-155.

8. Joy Manufacturing Company, Western Precipitation Division. Gas velocity and dust determination equipment. Catalog GV22. Los Angeles: Author.

9. Kimura, G. Emission control of flue-fed incinerators by use of air pollution control devices. Chicago: Department of Air Pollution Control, City of Chicago, November 1969. pp. 87-90.

10. PEDCo-Environmental Specialists, Inc. Administrative and technical aspects of source sampling for particulates. Contract No. CPA 70-124. Research Triangle Park, North Carolina: Environmental Protection Agency, May 1971.

11. Weisburd, M. (Ed.) Air pollution control field operations manual. A guide for inspection and enforcement. Washington, D. C.: U. S. Department of Health, Education, and Welfare; Public Health Service, Division of Air Pollution, 1962. PHSP #937. pp. 167-176.

Special Staffing Guidance

The task of determining the particulate concentration in the dry collector is similar in its general form to the suspended particulate concentration analysis (see D-37). The determination of the particulates trapped by the impingers is a relatively simple task requiring the manipulation of wet test analysis apparatus. Thus this task can be assigned to

a relatively inexperienced employee with moderate on-the-job training. This is especially true where the employee has already learned the suspended particulate concentration analysis and it is desired to introduce him to wet test analysis concepts, apparatus, and techniques.

Identification of the Constituents of Dust Particles

Task Overview

The objective of this task is to identify the constituents of dust particles so that the dust can be associated with a specific source. The dust sample is usually collected as a result of a citizen complaint or a routine patrol inspection.

The analysis of the dust sample consists of three parts:

1. An approximate identification is made through the use of a stereo microscope.
2. All compounds are identified using an X-ray defractometer.
3. A picture is taken of the sample for later use as a comparison standard.

Occupational Category: **Chemical Laboratory Technician or Equipment Technician**

Task Description

Perform initial identification using a stereo microscope:

1. Place the dust sample on a card or piece of filter paper and position it under a stereo microscope. (Choose the color of the card so as to provide a contrasting background for the sample.)
2. Adjust the light, focus, and magnification of the microscope until a clear image is obtained.
3. Identify the sample according to particle shape, texture, size, and color. Use standardized comparison samples as an aid to identification.
4. If the sample cannot be identified treat it physically or chemically under the microscope and observe the reaction:
    a. Use a magnet in order to detect the presence of iron.

b. Apply a drop of phenolpthalene to ascertain alkalinity which might indicate lime or cement dust.

c. Apply a drop of hydrochloric acid and look for a bubbling reaction which would indicate the presence of a carbonate.

Use X-ray defractometer to identify or verify dust constituents:

5. Grind a portion of the sample down to a size which is appropriate for X-ray analysis. Use a grinding machine such as the Wig L Bug.

6. Place a thin layer of the ground sample on a slide and insert it into the X-ray defractometer.

7. Start up the instrument, being sure to turn on cooling water and recorder, and adjust the voltage to the required setting. CAUTION: Wear X-ray badge.

8. Open the lead shield and start the instrument at 5° and allow the arm to run through the entire arc. Close the lead shield and shut down the instrument.

9. Remove the recorder chart and mark the degrees of arc on the chart.

10. Choose a peak on the chart and look up the angle (degrees of arc) on a table which describes the angle as a function of the distance between atoms of a compound. Obtain the value of the distance between atoms which corresponds to the angle.

11. Using the value of the distance between atoms ($\pm$ .01 Å), find the corresponding compounds on the table which lists compounds (and their X-ray defraction characteristics) as a function of distance between atoms. (This step narrows it down to a few pages of compounds.)

12. Based on the observations made under the stereo microscope, choose a compound from the table and note its peak strength. Compare the peak strength to that found on the chart. If it does not match, choose another compound.

13. If the peak strengths do match, note the other peaks caused by that compound. Check out each peak by working backwards through the tables, thus finding the corresponding angle and comparing it with the one on the chart. If all peak positions and strengths match, then the compound has been identified. If there is a discrepancy, choose another compound.

14. All peaks on the chart must be checked out until all constituent compounds are identified.

Photograph the sample:

15. Place a portion of the sample on a slide and place it under a polarizing microscope mounted with a Polaroid camera.

16. Turn on the light source and adjust voltage so that it corresponds with that indicated on the light meter.

17. Arrange the sample so as to best show all of the physical characteristics of the particles. This may require additional lighting or background.

18. Adjust the shutter time according to brightness and size of the particles.

19. Take the picture and treat the print with the chemicals provided by Polaroid.

Skill Requirements

1. Ability to adjust the light, focus, and magnification of a stereo microscope in order to obtain a clear image of a sample.

2. Ability to recognize the similarities and differences between the characteristics of a sample dust particle and the characteristics of a standard comparison sample. The important characteristics are particle shape, size, texture, and color.

3. Ability to determine how long a sample should be ground before it is ready for X-ray analysis.

4. Ability to spread the ground sample in a layer of the appropriate thickness on a slide.

5. Ability to accurately mark the recorder chart with the degrees of arc.

6. Ability to read the recorder chart interpreting the position and slope of the tracing in terms of the variables represented on the ordinate and absissa (i.e., the pollutant concentration over time or the strength of X-ray defraction as a function of beam angle).

7. Ability to find the compounds which correspond the the peak characteristics (location and strength) on the recorder chart, using the recorder chart and X-ray defraction tables. This involves a trial and error technique, enlightened by the knowledge gained from the preliminary analysis performed with the stereo microscope.

8. Ability to arrange the sample, the background, and lighting so as to best show the physical characteristics of the sample in a photograph taken with a polaroid camera mounted on a polarizing microscope.

9. Ability to determine the appropriate shutter speed in order to obtain an acceptable photograph of a dust sample, using a Polaroid camera mounted on a polarizing microscope. (The decision is based on the brightness and size of the dust particles.)

10. Ability to service and operate a chart recorder, including replacing chart roll, resupplying ink (if required), and adjusting the baseline (zero) point, line density, chart speed, and sensitivity range.

Knowledge Requirements

1. Knowledge of the apparatus required for the task and how it is configured.

2. Knowledge of the task procedure with all steps in their proper sequence.
3. Knowledge of the procedure for operating the stereo microscope.
4. Knowledge of the procedure for operating the grinding machine.
5. Knowledge of the procedure for operating the X-ray defractometer.
6. Knowledge of the procedure for using the X-ray defraction tables.
7. Knowledge of the procedure for operating the polarizing microscope with Polaroid camera.
8. Knowledge of the procedure for coating Polaroid prints.

Special Staffing Guidance

Since this task does not require skills and knowledge associated with wet test chemical analyses it could be as efficiently performed by either an Equipment Technician or a Chemical Laboratory Technician.

Maintenance of Laboratory Devices[1]

Task Overview

This task involves performance of various maintenance related activities on the instruments and equipment used in performing the laboratory analyses in order to maintain the accuracy and reliability of these devices. The instruments and equipment concerned include:

1. Colorimeters or spectrophotometers
2. Atomic absorption spectrophotometer
3. X-ray defractometers
4. Analytical balances
5. Drying ovens
6. Flowmeters
7. Vacuum pumps
8. Chart recorder

Occupational Category: Equipment Technician

Task Description

Due to the variety of kinds and types of devices included within this task it is not possible to state a step-by-step procedure for performing it. The task, however, does entail the following activities:

1. Installation, which involves unpacking, checking the device for damage, leveling, and making electrical and/or tubing connections.

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[1]The maintenance of the various laboratory devices has not been analysed in detail. Therefore, the skills and knowledge presented in the task package must be considered only as representative.

2. Servicing, which includes checking, cleaning, lubricating, adjusting, refilling and simple replacement operations performed on a scheduled basis.

3. Troubleshooting, i.e., identifying the cause of a malfunction through evaluation of symptoms and a systematic elimination of possible causes.

4. Repair, including repair, replacement, cleaning, and adjustment operations performed to rectify a specific malfunction.

5. Calibration, i.e., adjusting the performance of the device (e.g., a meter reading) to agree with some standard or developing a means of converting the performance of the device to the standard (e.g., development of a conversion chart or table).

6. Operation, including starting up, operating in various modes, and shutting off as necessary to perform the other activities.

Skill Requirements

1. Ability to read indicating devices, such as a thermometer, manometer, dry gas meter, and flow meter, and to interpret meter readings against a calibration plot as required.

2. Ability to prepare a calibration curve and compute the slope of the best fitting straight line, following a detailed step-by-step procedure. This may require the use of regression analysis by the least squares method.

3. Ability to read and interpret data from a table, psychometric chart, or a nomograph.

4. Ability to calibrate a sampling train or analyzer rotameter or flowmeter by making use of a wet test meter.

5. Ability to coordinate adjustment screw or hand knob movements with meter or chart recorder reading to quickly achieve and maintain the desired reading.

6. Ability to level instrument or analyzer during installation using a level, plumb, screw adjusters, and/or shims.

7. Ability to detect instrument damage caused by shipping, including dents, breakage, components knocked out of position, loose electrical and mechanical connections, and hairline cracks in glass parts.

8. Ability to detect leaks in hose, tubing, and piping connectors carrying liquids, gases, or vacuum using simple leak detection aids as appropriate.

9. Ability to disconnect and connect standard hose tubing and piping connectors without injury to threads and achieving leakproof connections. This includes the use of thread compound and teflon tape as appropriate.

10. Ability to disconnect and connect standard electrical connectors. This includes identifying leads to facilitate correct connection and achieving tight connections without damage to the leads or terminals.

11. Ability to correctly interpret function diagrams, wiring and tubing diagrams, simple electrical schematics, and trouble-shooting charts.

12. Ability to use electrical test instruments such as:

    1. AC/DC Voltmeter
    2. Ammeter
    3. Ohmmeter

    to achieve accurate circuit measurements without damage to the test instruments.

13. Ability to solder and unsolder electrical terminals making a good electrical and mechanical connection without shorting or grounding the connection or damaging circuit components.

Knowledge Requirements

For each device to be maintained:

1. Knowledge of the name and location of each part or component.
2. The location and function of each operating control.
3. The procedures for:
    a. Installation
    b. Servicing
    c. Troubleshooting
    d. Repair
    e. Calibration
    f. Operation
4. Knowledge of the tools, test instrumentation, supplies and materials necessary to perform the various procedures and where they are stored.

Special Staffing Guidance

The skills and knowledge required for this task are similar to those required for the operation and maintenance of the various analyzers described under Air Monitoring (see Volume E) and the same person could perform some, all, or some combination of these tasks.

Supervision of Laboratory Support Tasks

Task Overview

The function of lab task supervision is to direct, support, and evaluate the performance of the analysis and maintenance tasks carried out by the Chemical Laboratory Technicians and Equipment Technicians. The Supervisor further serves as an interface between the lab personnel and the users of lab services in other parts of the agency (e.g., air monitoring and source testing personnel) coordinating routine and special request analysis and advising users on proper sampling techniques.

Occupational Category: Chemist

Task Description

The procedure for supervising lab support tasks cannot be set down in a step-by-step sequence but involves various steps which are performed as often as the supervisor deems necessary in achieving timely and high quality results. These steps include:

1. Initiate the performance of the sampling and analysis activities or assure that the activities are performed as scheduled. This includes making work assignments.
2. Supervise the conduct of the activities, providing assistance as required.
3. Advise of special problems or contingencies which might affect when and how the activities are performed in a given instance (e.g., the need for non-scheduled sampling or the temporary presence of an interferent, the effect of which must be overcome or allowed for by special procedures).
4. Review the written report of the analyses for inconsistencies, mistakes, variations from the procedure, etc.

5. Work with professional level staff from other parts of the agency in planning and scheduling non-routine analysis (e.g., stack test analysis).

6. Assure that records of all analyses are in proper form, complete, and correctly filed.

7. Provide on-the-job training for all tasks in support of or in lieu of formal training provided elsewhere.

Skill Requirements

1. Ability to evaluate the quantity and quality of work produced by the staff and discriminate acceptable from unacceptable performance. This skill assumes the ability to use criteria of performance acceptability for all tasks supervised.

2. Ability to make work assignments and establish and maintain work schedules such that deadlines are met consistently.

3. Ability to develop work procedures which provide detailed step-by-step guidance in the performance of the analysis and instrument maintenance tasks.

4. Ability to document all procedures, findings, ideas, and decisions in writing which communicates clearly and completely to the intended audience, (e.g., Chemical Laboratory Technicians and Equipment Technicians).

5. Ability to effectively communicate verbally with Chemical Laboratory Technicians and Equipment Technicians concerning details of task performance.

6. Ability to co-operate with air monitoring and source testing personnel in planning tests so as to assure proper co-ordination of analysis and sample collection activities.

7. Ability to express technical theory and data in a concise, intelligent manner.

8. Ability to choose a sampling time length and flow rate in accordance with requirements for sample reliability and representativeness and to avoid overloading the capacity of the various reagents, filters, traps, etc., in the sampling train.

9. Ability to determine the extent to which equipment and instrument installation, troubleshooting, maintenance, and calibration should be performed by agency personnel given:

    a. The frequency with which these functions must be performed.

    b. The availability of the required skills and knowledge in-house or the cost of providing them through training or selection.

    c. The availability of the required test instruments, tools, and materials or the cost of providing them.

    d. The cost and delay associated with having some part of these functions performed outside the agency.

10. Ability to calculate a reasonable estimate of the total amount of moisture expected to be condensed from the gas sampled.

Knowledge Requirements

1. Knowledge of the capabilities and work loads of the personnel under his direction sufficient to permit the making of work assignments.

2. Knowledge of the sampling and analysis procedures, reagents, apparatus, and calculations for all analyses normally performed.

3. Knowledge of the theoretical background for the analyses and instruments of concern.

4. Knowledge of the service, maintenance, and calibration requirements of all lab instruments of concern.

5. Knowledge of the limitations associated with the analysis techniques being employed including:

   a. Range of ambient concentration of the pollutant for which the technique can be employed and means of modifying its sensitivity.

   b. Other constituents which interfere with the accuracy of the technique and methods for controlling their effect.

   c. Shelf life of the sample and correction for aged samples.

   d. Critical aspects of the technique (e.g., the need for nitrate-free water or the porosity requirements of the fritted bubbler) and means of assuring that these aspects are adequately provided for.

6. Knowledge of American Chemical Society reagent grade system and the effects of typical reagent impurities on the outcome of the analysis of concern.

7. Knowledge of factors surrounding the choice of appropriate sampling technique (e.g., the use of direct sampling, syringes, grab bottles, etc.).

8. Knowledge of each source which is pertinent to identifying analytical methodology, its use and location.

9. Knowledge of the meaning of the following terms which are used in the evaluation of a method:

   a. Validity

   b. Reliability

   c. Accuracy

   d. Precision

10. Knowledge of hazards involved with performing specific types of analytical tasks, such as:

    a. Presence of volatile or explosive chemicals

    b. Poisonous substances

    c. High temperatures or pressures

11. Knowledge of proper analytical laboratory procedures for obtaining valid results.

12. Knowledge of relevant factors sufficient to permit the development and periodic revision of a sampling schedule giving the times, durations, and locations of sampling for each ambient air sampling and analysis procedure to be employed. Relevant factors affecting the schedule include:

    a. Agency policy

    b. Current pollution levels

    c. Expected pollution levels

    d. Changes in the set of pollutants for which analyses are routinely performed

    e. Changes in sampling or analysis procedures

    f. Initiation of experimental monitoring programs

13. Knowledge of the relevant factors sufficient to permit the establishment and periodic revision of the sample flow rate to be maintained in the various sampling procedures. Relevant factors affecting flow rate include:

    a. Instrument manufacturers' recommendations

    b. Expected pollutant concentrations

    c. Efficiency of the sample and analyses processes

14. Knowledge of the analysis procedures sufficient to:

    a. Identify errors possible in each step of the procedure and their effect on the final outcome of the analysis.

    b. Identify critical steps in the procedure. A critical step is one in which

        (1) Errors are known to frequently occur

        (2) Little margin for error exists

        (3) Errors are likely to go undetected

    c. Revise procedures so as to reduce the possibility of error.

15. Knowledge of the chemical, electrical, and mechanical principles of operation of the various analysis instruments sufficient to:

    a. Identify instrument malfunctions which could go undetected and result in inaccurate read-out (to the extent not already documented in existing service manuals).

    b. Develop procedures for the timely discovery of such malfunctions.

    c. Identify the effects of incorrect instrument operation on instrument read-out.

16. Knowledge of the theoretical basis of stack testing procedures.

17. Knowledge of the procedure for determining the chemical composition (percent CO_2, CO, O_2, and N_2) of stack gas.

18. Ability to design a sampling train which is appropriate for a specific stack test with regard to the following criteria:

    a. Stack temperature.

    b. Particle size.

    c. Moisture content of gas.

    d. Corrosive nature of the gas.

    e. Type of analysis to be performed.

19 Knowledge of the procedure for calculating the expected sample weight.

20. Knowledge of the procedure for selecting an appropriate filtering media, given the expected sample weight, stack temperature, and the moisture content of the gas.

21. Knowledge of the efficiency, use restrictions, advantages, and disadvantages of each type of filtering media.

22. Knowledge of the procedure for selecting an appropriate condenser, based on the expected quantity of condensed moisture and cooling requirements for preventing further condensation.

23. Knowledge of the capacity, applicability, and advantages of different types of vacuum-producing devices, including a variety of pumps, blowers, and aspirators.

24. Knowledge of the information required and presentation format for reporting the results of a stack test.

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Special Staffing Guidance

The supervisor should be able to perform all the analyses and maintenance tasks performed by the Equipment Technicians and Chemical Laboratory Technicians under his supervision. But he needs, in addition,

familiarity with general laboratory techniques, the theoretical background of the analyses to be performed and the principles of operation of the laboratory instruments used. A Bachelor's Degree in Chemistry will provide many of the required skills and knowledge. It also should provide the background necessary for using technical literature and interaction with other Chemists to fill any skill and knowledge gaps.

Development of New Methods for the Analysis of Air Pollutants

Task Overview

Development of new analytical methods is a task which is found to occur frequently only in the older and larger agencies. It is a task which is difficult to describe in terms of steps, since it may involve different procedures adapted to solve different types of problems. For instance, a "new" method may simply involve a modification of an off-the-shelf instrument or it may involve an entirely unique approach to the measurement of air pollutants. The following task description identifies some of the typical requirements for method development.

Occupational Category: Chemist (Senior)

Task Description

1. Define the problem in terms of:
    a. What pollutant is to be measured.
    b. Requirements for precision and accuracy.
    c. Budget restrictions.
2. Investigate literature and personnel sources for existing methods of measurement pertaining to the pollutant. Possible sources include:
    a. Journals - air pollution control, analytical chemistry, occupational health.
    b. Technical magazines.
    c. Textbook materials.
    d. Environmental Protection Agency.
    e. Colleagues in other agencies and universities.
    f. Manufacturers of analytical instruments.

3. Evaluate each method and identify deficiencies according to the following criteria:

   a. Does the method provide a valid and reliable measurement of the pollutant?

   b. Does the method meet the requirements for precision and accuracy?

   c. Are there interferences from other pollutants?

   d. Is the method cost/effective and within the budget restrictions?

   e. Is the method extremely hazardous?

4. If there are no existing methods which can be used or modified in order to measure the pollutant, then construct an entirely new method. This method must be based on physical or chemical properties of the pollutant. which might include:

   a. Spectrographic emission.

   b. Flourescence.

   c. Molecular structure.

   d. Spectrophotometric absorption.

   e. Reactions with other compounds.

   f. Ionization.

   g. Chromatographic properties.

5. Choose the best suited method based on the evaluation criteria described in Step 3.

6. Obtain the necessary reagents and apparatus and place the method into a trial operation.

7. Observe the method in operation and identify unanticipated difficulties, e.g., inadequate filter media in a sampling train, or possibly an insufficient peak separation on a chromatogram.

8. Modify the method in order to compensate for the shortcomings identified.

9. Continue to test and modify the method until it meets the original requirements for measurement of the pollutant.

10. Develop a procedure for performing the new analysis such that a determination can be made of the problem which might arise in putting the technique into practice (e.g., special training required or unusual hazards present).

Skill Requirements

1. Ability to read, understand, interpret, and criticize technical documents which describe analytical chemistry laboratory techniques.

2. Ability to locate technical literature using abstracts and author or subject indexes.

3. Ability to effectively communicate with knowledgeable persons on the subject of analytical chemistry.

4. Ability to apply the knowledge of physical and chemical properties of pollutants, along with the knowledge of proper analytical procedure, to a new situation.

5. Ability to perform analytical laboratory procedures. This may include:

    a. Construction of apparatus.

    b. Sampling.

    c. Preparation of reagents.

    d. Operation of analytical instruments.

    e. Mathematical or statistical calculations.

    This skill is at least a composite of the skills required by the Chemical Laboratory Technician in the performance of the various analyses (see Pages D-4 through D-51).

6. Ability to identify and correct procedural difficulties which might affect the overall adequacy of a method.

7. Ability to develop a detailed step-by-step work procedure for the performance of the new analysis sufficient to permit the determination of the skills and knowledge required in carrying it out. Ability to evaluate the practicalities of the new technique based on:

    a. Required skills and knowledge not now possessed by laboratory staff.

    b. Unusual or extreme hazards.

    c. Complexity of the procedure.

    d. Overlong performance times.

    e. Steps in the procedure which have little margin for error (e.g., require extremely precise measurement).

Knowledge Requirements

1. Knowledge of each source which is pertinent to identifying analytical methodology, its use and location.

2. Knowledge of the meaning of the following terms which are used in the evaluation of a method:

    a. Validity

    b. Reliability

    c. Accuracy

    d. Precision

3. Knowledge of hazards involved with performing specific types of analytical tasks, such as:

    a. Presence of volatile or explosive chemicals.

    b. Poisonous substances.

    c. High temperatures or pressures.

4. Knowledge of chemical and physical properties of pollutants and how they can be manipulated and recorded so as to provide a unique indication of the presence and concentration of a specific pollutant. This requires extensive knowledge of:

    a. Atomic and molecular structure.

    b. Chemical reactions.

    c. Electro-chemical reactions.

    d. Reaction of atoms to excitation caused by burning, light, or X-rays.

    e. Other chemical or physical properties.

5. Knowledge of proper analytical laboratory procedures for obtaining valid results.

6. Knowledge of the limitations associated with the analysis techniques being employed including:

    a. Range of ambient concentration of the pollutant for which the technique can be employed and means of modifying its sensitivity.

    b. Other constituents which interfere with the accuracy of the technique and methods for controlling their effect.

    c. Shelf life of the sample and correction for aged samples.

    d. Critical aspects of the technique (e.g., the need for nitrate-free water or the porosity requirements of the fritted bubbler) and means of assuring that these aspects are adequately provided for.

7. Knowledge of American Chemical Society reagent grade system and the effects of typical reagent impurities on the outcome of the analysis of concern.

8. Knowledge of factors surrounding the choice of appropriate sampling technique (e.g., the use of direct sampling, syringes, grab bottles, etc.)

Special Staffing Guidance

This task requires a broad background of theory and technique in analytical chemistry much greater than that required for the day-to-day supervision of routine analyses. A thorough familiarity with existing techniques for analysis of air pollutants is, however, required to provide a basis for comparing the advantages and disadvantages of new methods. A familiarity with agency resources is also required to properly evaluate new methods. This implies that the person to whom this task is assigned should:

a. Be experienced in performing and supervising the standard analyses.

b. Have a thorough knowledge of the capabilities and limitations of the laboratory staff and equipment and budget restrictions affecting the possibility of increasing such capability.

c. Have advanced education and/or experience in the theory and techniques of analytical chemistry involving air pollutants.

It is for these reasons that the assignee be a senior Chemist.